Government of India, The Patent Office, 214, Acharya Jagadish Bose Road, Calcutta-17. Complete specification No. 143977 dated 29th April, 1976. Application and Provisional Specification No.701/Cal/1975 dated 8th April, 1975. Acceptance of the complete specification advertised on 4th March, 1978.

Index at acceptance - 70 C 4 + 5 [LVIII (5)]

International classification -C 23 b 9/02.

" Improvements in or relating to the process of hard anodising of aluminium and its alloys in sulphuric acid electrolyte using alternating current."

Council of Scientific and Industrial Research, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated uuder the Registration of Societies Act (Act XXI of 1860)

This is an invention by Balkunje Anantha Shenoi, Scientist, Venkataraman Balasubramanian, Senior Scientific Assistant, and Subbiah John, Senior Technical Assistant, all are Indian Nationals and employed in the Central Electrochemical Research Institute, Karaikudi-623006, Tamil Nadu, India.

The following specification describes the nature of this invention.

PRICE : TWO RUPEES

This invention relates to improvements in or relating to hard anodising of aluminium and its alloys in sulphuric acid electrolyte using alternating current.

Hitherto it has been proposed to use direct current or superimposed alternating current on direct current for hard anodising purposes with sulphuric acid.

The main drawbacks of the hitherto known processes are that i) hard anodising with direct current has a tendency for burning at the higher density regions unless the bath is provided with sufficient agitation and electrical contacts are good. Moreover, alloys containing over 3% copper or 7.5% silicon are difficult or impossible to treat by this process; ii) in the higher current density anodising processes operated with alternating current superimposed on direct current, a strong smell of hydrogen sulphide may be observed due to the cathodic reduction of sulphuric acid and consequent deposition of sulphur and its compounds inside the pores of the oxide film. Moreover, the use of both the types of currents and the necessary gadjets make the process cumbersome.

The main object of the present invention is to modify the alternating current sulphuric acid process so that the basic mechanism of electrolysis is shifted back in favour of the electrolysis of water rather than the reduction of sulphuric acid and thereby eliminating the drawbacks of alternating current in sulphuric acid. i.e. the formation and deposition of sulphur compounds inside the pores of the oxide film.

The main finding underlying the invention consists in hard anodising aluminium and its alloys using alternating current in an aqueous bath comprising the following : i) sulphuric acid ii) alkali metal salts such as sodium, potassium or lithium sulphate, potassium or sodium nitrate, sodium oxalate, sodium potassium tartrate , borax, sodium or potassium citrate, ammonium sulphate or nitrate.

The new result flowing from the new finding is that high current densities of the order of 8 A/dm$^2$ can be used without any burning, and alloys containing over 3% copper or 7.5% silicon which are impossible or difficult to treat by direct current process can be easily anodised without any smell of sulphuretted hydrogen and without any deposition of sulphur.

The present invention consists of a process for the hard anodising of aluminium and its alloys using alternating current in an aqueous bath consisting of sulphuric acid (d: 1.84) 5-20% v/v alkali metal salt 2-5% w/v at a temperature of -5 to +[illegible]°c for 45 to 90 minutes at a current density of 2.4 A/dm$^2$ to 8 A/dm$^2$ at a voltage of 20 to 80V. Hard anodising in such a bath gave hardness values of the order of 200 VPN to 500 VPN.

The novel features of this hard anodising process are that alternating current is used in place of direct current and alkali metals salts are used to modify the bath condition so produce a hard coating having the hardness as that produced with direct current.

The following typical examples are given to illustrate how the invention is carried out in actual practice but not to limit the scope of this invention.

EXAMPLE 1

2S aluminium plates (minimum 99% aluminium ) of [illegible] were mechanically polished, degreased with trichlorethylene, etched in alkaline solution, acid dipped and using alternating current (50 cycles,) hard anodising was carried out under the following conditions.

| | |
|---|---|
| Sulphuric acid | 15% v/v |
| Sodium sulphate | 5% wt/v |
| Temperature | $0^{o}$-$3^{o}C$ |
| Current | Alternating current |
| Current density | 40 $A/dm^2$ |
| Time | 60 minutes |
| Voltage | 30-70V |
| Thickness | 60 microns |

The plate got hard anodised and there was no hydrogen sulphide evolution. Hardness of the plate was 465 VPN

EXAMPLE 2

2S aluminium plates were cleaned as in Example 1 and using alternating current (50 cycles) hard anodising was carried out under the following conditions.

| | |
|---|---|
| Sulphuric acid | 15% v/v |
| Sodium tetra borate | 2% wt/v |
| Temperature | 1-4°C |
| Current density | 3.6 $A/dm^2$ |
| Voltage | 30-50 V |
| Time | 1 hour |
| Thickness | 58 microns |

The plates got hard anodised and there was no burning of the deposit. Hardness of the plate was 375-400 VPN

EXAMPLE 3

2S aluminium plates were cleaned as in Example 1 and using alternating current (50 cycles), hard anodising was carried out under the following conditions.

| | |
|---|---|
| Sulphuric acid | 15% v/v |
| Sodium oxalate | 2% wt/v |
| Temperature | 1-4°c |
| Voltage | 30-50V |
| Current density | 4.0 A/dm$^2$ |
| Time | 1 hour |
| Thickness | 50 microns |

The plates got hard anodised and there ware no etching. Hardness of the plate was 390-400 VPN

EXAMPLE 4

2S aluminium plates were cleaned as in Example and using alternating current(50 cycles), hard anodising was carried out under the following conditions .

| | |
|---|---|
| Sulphuric acid | 15% v/v |
| Potassium sulphate | 3% wt/v |
| Temperature | 1-4°c |
| Voltage | 30-60 V |
| Current density | 6.0 A/dm$^2$ |
| Time | 1 hour |
| Thickness | 70 microns |

The plates got hard anodised and hardness was470 VPN.

EXAMPLE 5

2S aluminium plates were cleaned and hard anodised under the following conditions using alternating current (50 cycles)

| | |
|---|---|
| Sulphuric acid | 15% v/v |
| Sodium potassium tartrate | 3% w/v |
| Temperature | 1-3°c |
| Current density | 4.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | 30-60 V |
| Time | 1 hour |
| Thickness | 57 microns |

The plates got hard anodised and the hardness was 400 VPN.

EXAMPLE 6

2S aluminium plates werecleaned and hard anodising was carried out using alternating current (50 cycles) under the following conditions.

| | |
|---|---|
| Sulphuric acid | 10% v/v |
| Sodium nitrate | 3% w/v |
| Current density | 4.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | 30-60 V |
| Time | 1 hour |
| Thickness | 60 microns |

The plates got hard anodised and its hardness was 390-400 VPN

EXAMPLE 7

LM4 aluminium cast alloy (containing 2-4% copper and silicon 4-6% plates were cleaned and hard anodised under the following conditions

| | |
|---|---|
| Sulphuric acid | 15% v/v |
| Potassium bisulphate | 3% w/v |
| Temperature | -1 to 4$^o$c |
| Current density | 6.0 A/dm$^2$ (AC 50 cycles) |
| Voltage | 30-60 V |
| Time | 1 hour |
| Thickness | 70 microns |

The plate got hard anodised and its hardness value was 300 VPN.

EXAMPLE 8

HE-15 aluminium alloy plates (containing 4.25% Copper, 0.75% Silicon, 0.75% Manganese, 0.5% magnesium, rest aluminium) were cleaned and hard anodised under the following conditions.

| | |
|---|---|
| Sulphuric acid | 15% v/v |
| Potassium Sulphate | 3% w/v |
| Temperature | -1 to 4$^o$c |
| Current density | 6.0 A/dm$^2$ (AC 50 cycles) |
| Voltage | 25-60 V |
| Time | 1 hour |
| Thickness | 72 microns |

The plate got hard anodised and its hardness value was 300 VPN.

EXAMPLE 9

2S aluminium plates were cleaned and hard anodised using alternating current (50 cycles) under the following conditions.

| | |
|---|---|
| Sulphuric acid | 15% |
| Ammonium nitrate | 3% |
| Temperature | -1 to 4$^o$c |
| Current density | 4.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | 30-50 V |
| Time | 1 hour |
| Thickness | 56 microns |

The plate got hard anodised and its hardness value was 200 VPN

EXAMPLE 10

2S aluminium plates were cleaned and hard anodised under the following conditions using alternating current (50 cycles )

| | |
|---|---|
| Sulphuric acid | 12.5% |
| Ammonium sulphate | 3% |
| Temperature | -1 to 4°c |
| Current density | +.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | 25-55 V (30-55V) |
| Time | 1 hour |
| Thickness | 60 microns |

The plate got hard anodised and its hardness was 210 VPN.

EXAMPLE 11

26S aluminium plates (containing 4.25% copper, rest aluminium were cleaned and hard anodised using alternating current (50 cycles) under the conditions cited in Example 1. The hardness of the plate was 400 VPN and its thickness was 69 microns.

EXAMPLE 12

26 S aluminium plates were cleaned and hard anodised using alternating current (50 cycles ) under the following conditions.

| | |
|---|---|
| Sulphuric acid | 12.5 % |
| Potassium nitrate | 3% |
| Temperature | -1 to 4°c |
| Current density | 4.8 A/dm$^2$ |
| Voltage | 25-55V |
| Time | 1 hour |
| Thicknes | 60 microns. |

The plate got hard anodised and hardness value was 400 VPN

EXAMPLE 13

2S aluminium plates were cleaned and hard anodised with the use of alternating current (50 cycles) under the following conditions.

| | |
|---|---|
| Sulphuric acid | 10% |
| Sodium or potassium acetate | 3% |
| Temperature | 0-4 °c |
| Current dens ity | 4.8 A/dm$^2$ |
| Voltage | 30-60 V |
| Time | 1 hour |
| Thickness | 56 microns |

The plate got hard anodised and its hardness value was 250 VPN

EXAMPLE 14

3S aluminium (containing 1.2% Mn, rest aluminium) were cleaned and hard anodised under the following conditions.

| | |
|---|---|
| Sulphuric acid | 10% |
| Lithium sulphate | 3% |
| Temperature | 0-4°c |
| Current density | 4.8 A/dm$^2$(A.C 50 cycles) |
| Voltage | 35-60 V |
| Time | 1 hour |
| Thickness | 55 microns |

The plate got hard anodised and the hardness value was 300 VPN.

Example 15

3S aluminium plates were cleaned and hard anodised using alternating current (50 cycles) under the following conditions

| | |
|---|---|
| Sulphuric acid | 15% |
| Sodium sulphate | 3% |
| Temperature | 0-4°c |
| Current density | 8 A/dm$^2$ |
| Voltage | 35-65V |
| Time | 1 hour |
| Thickness | 80 microns |

The plate got hard anodised and the hardness was 600 VPN.

The following are among the main advantagesof the invention:

1. The method offers the opportunity of anodising in a 'box ' of electrolyte with no cathode plates or other encumbrances. This willresult in the possibility of high volume production anodising, since the current loading of an anodiser can be near ideal all the metal in the tank is to be anodised and the electrolyte volume can be fully utilised.

2. If high current densities of the order of 4.8 A/dm$^2$ can be used in a suitable electrolyte, the chances of burning are limited and again work can be processed faster through the anodising tank, either by passing more loads through or by ensuring complete current utilisation by optimum loading.

3. The cost of electrical equipment for aleternating current anodising ✱ much cheaper than direct current, since all that is required is a stepdown transformer and voltage regulator. Switch gear is also muchlighter in construction.

4. Alloys which are normally regarded as difficult to anodise i.e high copper and silicon wrought alloys, most castings etc . can be easily processed because of the tendency of alternating current to reduce the change of burning at high current densities and voltages;

5. Owing to the natural cleaning power of alternating current where large volumes of hydrogen and oxygen are alternattely evolved, the necessity of pretreatment of difficult alloys, i.e those containing copper and silicon, can be kept to minimum so that the occurence of metallurgical defects often revealed by chemical pretreatment can be reduced.

Dated this 29th day of March 1975

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ASST PATENT OFFICER

COUNCIL OF SCIENTIFIC & INDISTRIAL RESEARCH

143977

THE PATENTS ACT, 1970

COMPLETE SPECIFICATION

SECTION 10.

"Improvements in or relating to the process of hard anodising of aluminium and its alloys in sulphuric acid electrolyte using alternating current."

Council of Scientific and Industrial Research, Rafi Marg, New Delhi-1, India, an Indian registered body incorporated under the Registration of Societies Act (Act XXI of 1860)

This is an invention by Balkunje Anantha Shenoi, Scientist, Venkataraman Balasubramaniam, Senior Scientific Assistant, and Subbiah John, Senior Technical Assistant, all are Indian Nationals and employed in the Central Electrochemical Research Institute, Karaikudi 623006 Tamil Nadu, India.

The following specification particularly describes and assertains the nature of this invention and ithe manner in which it is to be performed.

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This invention relates to improvements in or relating to hard anodising of aluminium and its alloys in sulphuric acid electrolyte using alternating current.

Hitherto it has been proposed to use direct current or superimposed alternating current on direct current for hard anodising purposes with sulphuric acid.

The main drawbacks of the hitherto known processes are that (i)hard anodising with direct current has a tendency for burning at the high current density regions unless the bath is provided with sufficient agitation and electrical contacts are good. Moreover, alloys containing copper or silicon are difficult or impossible to treat by this process; (ii) in the higher current density anodising processes operated with alternating current superimposed on direct current, a strong smell of hydrogen sulphide may be observed due to the cathodic reduction of sulphuric acid and consequent deposition of sulphur and its compounds inside the pores of the oxide film. Moreover, the use of both the types of currents and the necessary gadgets make the process cumbersome.

The main object of the present invention is to obviate the above disadvantages by employing a modified $H_2SO_4$ bath so that the basic mechanism of electrolysis is shifted back in favour of the electrolysis of water rather than the reduction of sulphuric acid and thereby eliminating the drawbacks of the existing process, i.e., the formation and deposition of sulphur compounds inside the pores of the oxide film. Further the addition agents added to the anodising bath has no deliterious effect and it does not decompose in the bath.

The main finding underlying the invention consists in hard anodising of aluminium and its alloys using alternating current from an aqueous electrolyte containing 2.5 to 20% W/V sulphuric acid and 0.5 to 10% W/V of one or more of the addition agents such as alkali metal acetates, sulphates, nitrates, borates, tartarates and oxalates of sodium, potassium, lithium and ammonium at temperatures between -5 to +5°C, using current densities of upto 8 A/dm$^2$, for 30 to 120 min. to produce an oxide coating of thickness 25 to 80 microns, to give hardness values of upto 600 VPN.

The result flowing from the new finding is that high current densities of the order of 8 A/dm$^2$ can be used without any burning and alloys containing over 3% copper or 7.5% silicon which are impossible or difficult to treat by direct current process can be anodised easily without any smell of sulphuretted hydrogen and without any deposition of sulphur.

The present invention consists of a process for hard anodising aluminium and its alloys using alternating current in sulphuric acid electrolyte which consists in polishing, degreasing, cleaning and hard anodising aluminium and its alloys in 2.5-20% v/v sulphuric acid (d: 1.84) electrolyte containing addition agents such as alkali metal sulphates, oxalates, nitrates, citrates, acetates, tartarates and borates in the range of 0.5 to 10% W/v at a temperature of -5 to +5°C for 45-90 minutes, at current densities upto 3A/dm$^2$, at voltages of 20 to 60 V, to give hardness values of the order of 200 to 600 VPN.

The novel features of this hard anodising process are that alternating current is used in place of direct current or A.C. superimposed D.C. and alkali metal salts are used to modify the bath bondition so as to produce a hard and thick coating in a shorter duration since high C.D. is employed.

The following typical examples are given to illustrate how the invention is carried out in actual practice but not to limit the scope of this invention:

EXAMPLE 1

2 S aluminium plates (minimum 99% aluminium) of size 5 x 7.5 cm were mechanically polished and buffed to remove the surface defects like scratches, corrosion spots etc. and degreased with a solvent such as trichloroethylene to remove polishing compound and greases from the surface and alkaline etched in 5% W/V sodium hydroxide for further cleaning of the surface and dipped in 10% V/V nitric acid for removing the sludge formed during alkaline etching (with an intermediate washing) and the cleaned panel is hard anodised under the following conditions using alternating current:

| | |
|---|---|
| Sulphuric acid | : 15% v/v |
| sodium sulphate | : 0.5% W/v |
| Temperature | : 0° - 3°C |
| Current | : Alternating current |
| Current density | : 40 A/dm$^2$ |
| Time | : 60 minutes |
| Voltage | : 30-70V |
| Thickness | : 60 microns |

The plate got hard anodised and there was no hydrogen sulphide evolution. The presence of sulphur in the oxide film was tested by dipping the specimen in lead acetate solution which did not turn into brown or black showing the absence of sulphur. Hardness of the plate was 465 VPN.

EXAMPLE 2

2S aluminium plates were cleaned as in Ex.1 and using alternating current (50 cycles) hard anodising was carried out under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 2.5 v/v |
| Sodium tetra-borate | : 2% w/v |
| Temperature | : 1-4°C |
| Current density | : 3.6 A/dm$^2$ |
| Voltage | 30-50V |
| Time | : 1 hour |
| Thickness | : 58 microns |

The plates got hard anodised and there was no burning of the deposit. Hardness of the plate was 375-400 VPN.

EXAMPLE 3

2S aluminium plates were cleaned as in Ex.1 and using alternating current(50 cycles), hard anodising was carried out under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 15% v/v |
| Sodium oxalate | : 2% w/v |
| Temperature | : 1-4°C |
| Voltage | : 30-50V |
| Current density | : 4.0 A/dm$^2$ |
| Time | : 1 hr. |
| Thickness | : 50 microns |

The plates got hard-anodised and there was no etching. Hardness of the plate was 390-400 VPN.

EXAMPLE 4

2S aluminium plates were cleaned as in Ex.1 and using alternating current (50 cycles), hard anodising was carried out under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 5% v/v |
| Potassium sulphate | : 3% w/v |
| Temperature | : 1-4°C |
| Voltage | : 30-60 V |

| | |
|---|---|
| Current density | : 6.0 A/dm$^2$ |
| Time | : 1 hr. |
| Thickness | : 70 microns |

The plates got hard anodised and hardness was 470 VPN.

**EXAMPLE 5**

2S aluminium plates were cleaned as in example 1 and hard anodised under the following conditions using alternating current (50 cycles):

| | |
|---|---|
| Sulphuric acid | : 7.5% v/v |
| Sodium potassium tartarate | : 3% w/v |
| Temperature | : 1-3°C |
| Current density | : 4.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | : 30-60 V |
| Time | : 1 hr. |
| Thickness | : 57 microns |

The plates got hard anodised and the hardness was 400 VPN.

**EXAMPLE 6**

2S aluminium plates were cleaned as in example 1 and hard anodising was carried out using alternating current (50 cycles) under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 20% v/v |
| Sodium nitrate | : 10% w/v |
| Current density | : 4.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | : 30-60 V |
| Time | : 1 hr. |
| Thickness | : 60 microns |

The plates got hard anodised and its hardness was 390-400 VPN.

**EXAMPLE 7**

LM 4 aluminium cast alloy(containing 2-4% copper and silicon 4-6%) plates were degreased and hard anodised under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 15% v/v |
| Potassium bisulphate | : 3% w/v |
| Temperature | : -1 to 4°C |
| Current density | : 6.0 A/dm$^2$ (AC 50 cycles) |
| Voltage | : 30-60 V |
| Time | : 1 hour |
| Thickness | : 70 microns |

The plate got hard anodised and its hardness value was 300 VPN.

## EXAMPLE 8

HE-15 aluminium alloy plates (containing 4.2% copper, 0.65% silicon, 0.75% manganese, 0.5% magnesium, rest aluminium) were degreased and hard anodised under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 15% v/v |
| Potassium sulphate | : 3% w/v |
| Temperature | : -1 to 4°C |
| Current density | : 6.0 A/dm$^2$ (AC 50 cycles) |
| Voltage | : 25-60 V |
| Time | : 1 hour |
| Thickness | : 72 microns |

The plate got hard anodised and its hardness value was 300 VPN.

## EXAMPLE 9

2S aluminium plates were cleaned as in example I and hard anodised using alternating current (50 cycles) under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 15% |
| Ammonium nitrate | : 3% |
| Temperature | : -1 to 4°C |
| Current density | : 4.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | : 30-50 V |
| Time | : 1 hour |
| Thickness | : 56 microns |

The plate got hard anodised and its hardness value was 200 VPN.

## EXAMPLE 10

2S aluminium plates were cleaned as in example 1 and hard anodised under the following conditions using alternating current (50 cycles):

| | |
|---|---|
| Sulphuric acid | : 1.5% |
| Ammonium sulphate | : 3% |
| Temperature | : -1 to 4°C |
| Current density | : 4.8 A/dm$^2$ (AC 50 cycles) |
| Voltage | : (30-55 V) |
| Time | : 1 hour |
| Thickness | : 60 microns |

The plate got hard anodised and its hardness value was 210 VPN.

## EXAMPLE 11

26S aluminium plates (containing 4.2% copper, rest aluminium) were degreased and hard anodised using alternating current (50 cycles) under the conditions cited in Ex.1. The hardness of the plate was 400 VPN and its thickness was 69 microns.

## EXAMPLE 12

26S aluminium plates were degreased and hard anodised using alternating current (50 cycles) under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 12.5% |
| Potassium nitrate | : 3% |
| Temperature | : -1 to 4°C |
| Current density | : 4.8 $A/dm^2$ |
| Voltage | : 25 - 55V |
| Time | : 1 hour |
| Thickness | : 60 microns |

The plate got hard anodised and hardness value was 400 VPN.

## EXAMPLE 13

2S aluminium plates were cleaned as in example 1 and hard anodised with the use of alternating current (50 cycles) under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 10% |
| Sodium or potassium acetate | : 3% |
| Temperature | : 0-4°C |
| Current density | : 4.8 $A/dm^2$ |
| Voltage | : 30-60 V |
| Time | : 1 hour |
| Thickness | : 56 microns |

The plate got hard anodised and its hardness value was 250 VPN.

## EXAMPLE 14

3S Aluminium(containing 1.2% Mn, rest aluminium) were cleaned as in example 1 and hard anodised under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 10% |
| Lithium sulphate | : 3% |
| Temperature | : 0-4°C |
| Current density | : 4.8 $A/dm^2$ (AC 50 cycles) |
| Voltage | : 35-60 V |
| Time | : 1 hour |
| Thickness | : 55 microns |

The plate got hard anodised and the hardness value was 300 VPN.

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EXAMPLE 15

3S aluminium plates were cleaned as in example 1 and hard anodised using alternating current (50 cycles) under the following conditions:

| | |
|---|---|
| Sulphuric acid | : 15% |
| Sodium sulphate | : 3% |
| Temperature | : 0-4°C |
| Current density | : 8 $A/dm^2$ |
| Voltage | : 35-65 V |
| Time | : 1 hour |
| Thickness | : 80 microns |

The plate got hard anodised and the hardness was 600 VPN.

The following are the main advantages of the invention:

1. This process of hard anodising aluminium and its alloys using alternating current in modified sulphuric acid electrolyte offers the opportunity of anodising in a tank of electrolyte wherein both the electrodes are aluminium. Hence the entire volume of the electrolyte can be effectively utilised for anodising more area of the work with maximum current loading.

2. This modified sulphuric acid bath when used for hard anodising aluminium and its alloys using alternating current, there will be no burning due to high current densities(8$A/dm^2$), and hence the work can be processed quickly or more work can be processed in a given time.

3. The cost of electrical equipment for alternating current anodising is much cheaper than direct current, since all that is required is a step down transformer and voltage regulator. Switchgear is also much lighter in construction.

4. Alloys which are normally regarded as difficult-to-anodise, i.e., high copper and silicon wrought alloys, most castings etc. can be easily processed because of the tendency of alternating current to reduce the chance of burning at high current densities and voltages.

5. Owaing to the natural cleaning power of alternating curr. where large volumes of hydrogen and oxygen are alternately evolved, the necessity of pretreatment of difficult alloys, i.e., those containing copper and silicon can be kept to minimum so that the occurrence of metallurgical defects often revealed by chemical pretreatment can be reduced.

A process for hard anodising aluminium and its alloys using alternating current in sulphuric acid electrolyte consists in polishing and buffing, degreasing, cleaning and hard anodising in 2.5-20% v/v sulphuric acid electrolyte containing addition agents such as alkali metal sulphates, oxalates, nitrates, citrates tartarates and borates in the range of 0.5 to 10% w/v using alternating current.

WE CLAIM

A process for hard anodising of aluminium and its alloys containing silicon and copper using alternating current in sulphuric acid electrolyte which comprises the steps of mechanical polishing and buffing, degreasing with trichloroethylene, alkalin etching and dipping in acid, with an intermediate washing and hard anodising in an aqueous electrolyte containing 2.5 to 20% v/v sulphuric acid and 0.5 to 10% w/v of one or more of the addition agents such as alkalimetal acetates, sulphates, nitrat borates, tartarates and oxalates of sodium, potassium, lithium and ammonium at temperatures between -5 to +5°C using current densities of upto 8 $A/dm^2$ for 30 to 120 min to produce a hard oxide coating of 25 to 80 microns thickness to give hardness values of upto 600 VPN.

Dated this 26th day of April, 1976

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